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Ultra High Vacuum Instrumentation Development Studies

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Abstract

Attempts at using the Spindt-type molybdenum field emitter arrays in the extractor gauge (EG) and residual gas analyzer (RGA) are presented in this paper. The sensitivity of the field emitter gauge (FEG) is as high as 11 Torr<sup>-1</sup>. There is an excellent measurement linearity for FEG and the linear deviation is smaller than 10% in the pressure range of 10<sup>-11</sup>-10<sup>-6</sup> Torr. We achieved quite stable sensitivities for nitrogen, helium and hydrogen with the field emitter RGA (FERGA) below 10<sup>-7</sup> Torr. The slightly reduced emission current and sensitivity, after

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long-term operation, are of concern and need to be addressed. Residual gas spectra indicate that when using field emitters, the electron stimulated desorbtion (ESD) ions (O+, F+ and Cl+) are lower than those of hot filament generated spectra.

#### 1. Introduction

UHV and XHV measurements are generally affected by the thermally desorbed ions due to hot filaments. The ESD ions also considerably affect the UHV and XHV measurements as a result of high emission currents used in these vacuum gauges for increasing the ion signals. In addition, the chemical effects and the x-ray limit are the other sources of pressure measurement errors<sup>1</sup>. The effect of turning on of the hot filament on system pressure has recently been demonstrated very elegantly<sup>2</sup>. Several people have tried field emitter electron sources in pressure measurement instrumentation<sup>3,4</sup>.

We have modified a Leybold Inficon EG and a 100 AMU SRS residual gas analyzer (RGA) by replacing the hot filament with Spindt type field emitters<sup>5</sup>. In this paper, we report measurements on the sensitivity and the linearity of these two instruments. In addition, the ESD related differences between the hot filament and the field emitter electron sources in the UHV regime are also discussed.

#### 2. Experimental

The hot filaments of two Leybold Inficon extractor gauges are replaced with the Spindt field emitters. A molybdenum field emitter array (10000) of 0.266 in.-dia is fixed in a 0.75

in.-dia. cathode mount. Two types of field emitter extractor gauge (FEG) are fabricated. In the first one the field emitter array is mounted beside the cylindrical anode grid (side FEG) by simply replacing the existing hot filament. In the second one the field emitter array is mounted above the grid (top FEG) and the extended hot filament pin outs serve as the mounts for the field emitter as shown in Fig. 1a. In the case of SRS RGA the field emitter is mounted close to the anode grid structure and is shown in Fig. 1b. The hot filament of the RGA is left intact and the electronic controller is modified such that we can switch either the field emitter or hot filament on at any given time. The distance between the field emitter array and the grid top is optimized by the Semion 3D computer simulation for the top FEG and FERGA. FERGA spectra are always taken with the channel electron multiplier (CEM) due to very small ion currents.

Evaluation of the modified instruments is carried out in the Jefferson Lab vacuum gauge calibration facility. The facility is designed and fabricated as per ISO/DIN 3567 standard. A Leybold Inficon IE 514 extractor gauge is selected as reference gauge in the UHV pressure range. The extractor gauge itself is calibrated with an MKS spinning rotor gauge in the pressure range  $10^{-5}$  -  $10^{-7}$  Torr. Four Keithley electrometers are used to measure various electrode currents. Semion 3D ion modeling software from Scientific Instrument Services, Inc. has been used for designing the optimum gauge structure as well as the analysis of the data.

#### 3. Results and discussion

(1) FEG

The nitrogen sensitivities of both the top and side FEG's are shown in Fig. 2 as a function of the anode potential. The maximum sensitivity of the top FEG is ~ 8 Torr-1 (with 500 V anode potential) while the side FEG achieved a sensitivity of 11 Torr-1 (after optimization but not shown in the figure). As can be expected the sensitivity improves with the increasing anode potential as a result of the enhanced electron energy. Since the x-ray current increases linearly with the increase of anode potentials<sup>6</sup>, generally high sensitivity is traded for the low x-ray background by using low anode potential. Semion 3D computer analysis of the sensitivities data explains the high sensitivity of the side FEG in comparison to the top FEG. The sensitivity enhancement of the side FEG appears to be contributed by two factors. First, the average electron path length of the side FEG is longer. Second, the electron velocity is almost constant within the anode grid; therefore the ionization efficiency is higher than the top FEG in which the electron energy is lower than 100 eV during about 30% of electron trajectory.

The normalized ion current of the top FEG is presented in Fig. 3 as a function of nitrogen partial pressure in the pressure range  $10^{-11}$ - $10^{-5}$  Torr. The linear response is quite evident over the operating pressure range. The deviation from linearity is smaller than 10% over the entire pressure range. The side FEG sensitivities for nitrogen and hydrogen are shown for various electrode potential and electron emission current combinations (anode voltage Ua, reflector voltage Ur, cathode voltage Uk and the gate voltage Ug) in Figs. 4 a and b. The sensitivities remain fairly constant with different emission currents (in the range 33- $100~\mu$ A) and at the noted fixed electrode potentials. The side FEG nitrogen sensitivities and the helium and hydrogen relative sensitivity factors are listed with the corresponding values

for the IE 514 extractor gauge in table 1. The helium and hydrogen relative sensitivity factors are almost constant when increasing the anode potentials from 250V to 500V.

The operation of the field emitter at pressures higher than  $1 \times 10^{-7}$  Torr appears to induce a reduction of emission current and this effect is graphically presented in Fig. 5. The emission current declined after an increase in the nitrogen partial pressure to  $2.5 \times 10^{-7}$  Torr for 10 min. The emission current of  $5.42 \times 10^{-5}$  A dropped to  $3.85 \times 10^{-5}$  A and only returned to 92% of its original value in 48 hours and after the pressure was restored to  $10^{-10}$  Torr. The complete recovery of the emission current may probably take a very long time. This might be due to microscopic changes of the tip geometry. The sputtering rate will go up when vacuum deteriorates and this is likely to lead to tip melting via resistive heating and eventual deterioration of the emission current. Additionally, we have seen an emission current increase after the field emitter is operated in hydrogen atmosphere. This emission current enhancement appears to be due to the reduced work function of the emitter tips in the presence of hydrogen  $^{8,9}$ .

After prolonged testing of FEG's with nitrogen, helium and hydrogen between 10<sup>-10</sup>10<sup>-6</sup> Torr, the sensitivities were found to have declined slightly. Fig. 6 shows the sensitivity variations under different test conditions and with various electrode voltage combinations.

The sensitivity decreased ~ 10% after 38 days of operation (test 2). Sensitivities return to their original values after the field emitter is exposed to air for 48 hours following switching it back on after lowering the pressure to the original operating range (test 3). We believe that a decrease in the work function of the field emitter due to readsorption of hydrogen and other chemical active atoms contributed to this correction.

### (2) FERGA

The normalized sensitivities and emission currents for nitrogen, helium and hydrogen are presented in Fig. 7 in the pressure range  $10^{-10}$  to  $10^{-5}$  Torr. The sensitivities and emission currents are very stable and their uncertainties are less than 10% for hydrogen below  $1\times10^{-7}$  Torr. The emission currents begin to decrease from middle  $10^{-9}$  Torr and lower for nitrogen and helium, and the current drops over 30% for nitrogen when pressure reaches above  $5\times10^{-7}$  Torr.

Fig. 8 shows the typical residual mass spectra with the FERGA and hot filament RGA. The spectra are obtained at the same pressure  $(2.3\times10^{-10} \text{ Torr})$ , electron emission current  $(5.3\times10^{-5} \text{ A})$ , and electron and ion energies. We compare the spectral features as follows:

- a). Hydrogen (mass 2) and water vapor (masses 17 and 18) have identical partial pressure readings for both kinds of cathodes.
- b). Peaks 28 (CO) and 44 (CO<sub>2</sub>) are lower for FERGA. The CO and CO<sub>2</sub> for the hot filament are mainly higher because the thoria coated iridium filament can become a source of oxygen for producing CO and CO<sub>2</sub> in the presence of hydrogen<sup>10</sup>.
- c). ESD ions O+ (mass 16), F+ (mass 19) and Cl+ (mass 35 and 37) are lower in the field emitter mode. One possible reason for this is the influence of the field emitter geometry and its location with respect to the ionization source. For the FERGA, the field emitter array (0.266-in.-dia.) is fixed in a 0.75-in.-dia. cathode mount and it is positioned only 0.2 in. away from the grid top. According to the ion modeling software simulations, the trajectories of the ions that are produced near the top grid are seriously influenced by the

potential are 10 V and 90 V lower than the anode grid potential respectively. A large part of the ions, including ESD ions, are attracted to the field emitter array and join in the gate current and the emitter cathode current. The computer simulation shows that most of the electrons stopped on or near the top grid, so most of the ESD ions come from the top grid area and some of such ESD ions are attracted to the field emitter array. This effect will decrease the collected ESD ions by the RGA ion detection system. Hence the ESD signals for the FERGA are smaller than those of the hot filament RGA.

The FEG results also show that the measurement linearity's of the Top FEG are better than those of the Side FEG in the pressure range 10<sup>-11</sup> Torr. Such a reduced ESD ions

d). When increasing electron energy, peaks 16(O+) and 19(F+) increase for both RGAs, whereas peaks 35 and 37(Cl+) increase for the FERGA only and are not almost affected in the case of the conventional hot filament RGA.

effect should be confirmed in future XHV research.

## 4. Summary

The UHV characteristics of the field emitter extract gauge (FEG) and RGA (FERGA) are presented in this paper. The sensitivities of the top and side FEG's are 8 Torr<sup>-1</sup> and 11 Torr<sup>-1</sup> respectively. Higher sensitivity can in principle be obtained by increasing the anode potential. But the x-ray current also will increase approximately linearly with the increasing anode potential. FEG's have excellent measurement linearity in the pressure range 10<sup>-11</sup> - 10<sup>-6</sup> Torr. The small emission current is beneficial in reducing the positive ion space charge

repulsion so as to improve high pressure measurement linearity. The emission current will decline irreversibly when the field emitter is operated over  $1 \times 10^{-7}$  Torr. It appears that the gauge sensitivity decreases after long operation. However, exposing the field emitter to air seems to recover the sensitivity value because the work function of the emitter tip is improved by some chemically active atoms such as H. The turning on of the field emitter will produce a much smaller and a shorter period pressure rise in comparison to the hot filament.

The stability of the FERGA sensitivity is satisfactory below 10<sup>-7</sup> Torr. ESD ions are the most troublesome source for UHV/XHV measurements<sup>11,12</sup>. The residual mass spectra of the FERGA indicate that ESD ions are lower than those of the hot filament RGA. This reduction is probably caused by the attraction of the ESD ions to the field emitter. The top field emitter structure is expected to have a definite advantage for UHV/XHV measurement when the effect of reducing collected ESD ions is further confirmed in XHV.

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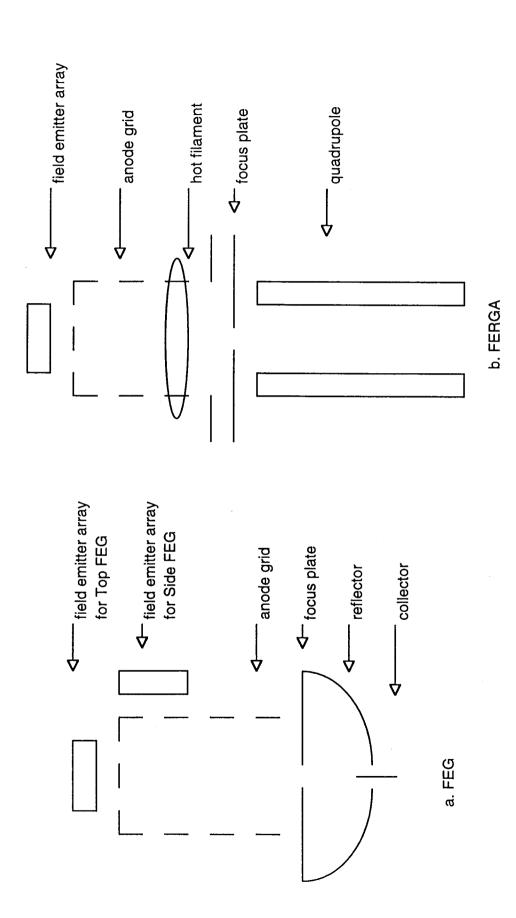


Fig.1 Schematic setups of the field emitter Extractor Gauge (FEG) and the field emitter RGA (FERGA)

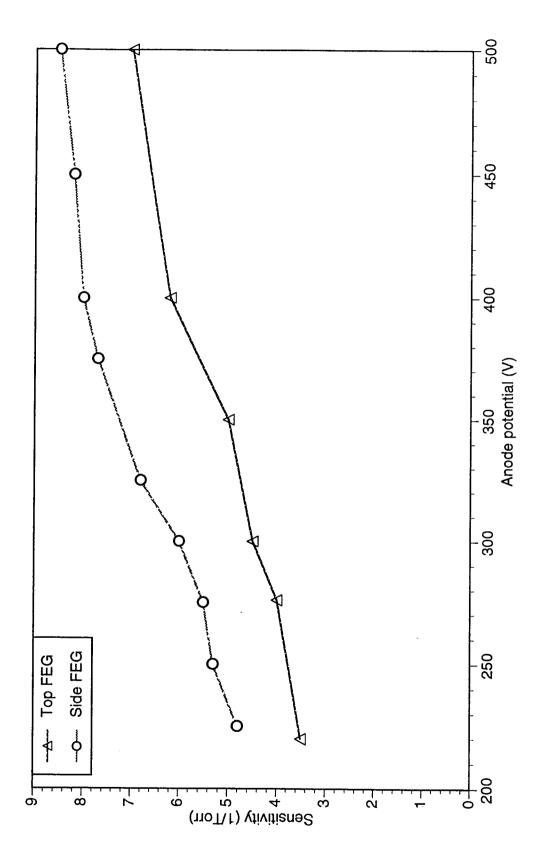


Fig.2 Sensitivity increase with the anode potential (electron energy) increase

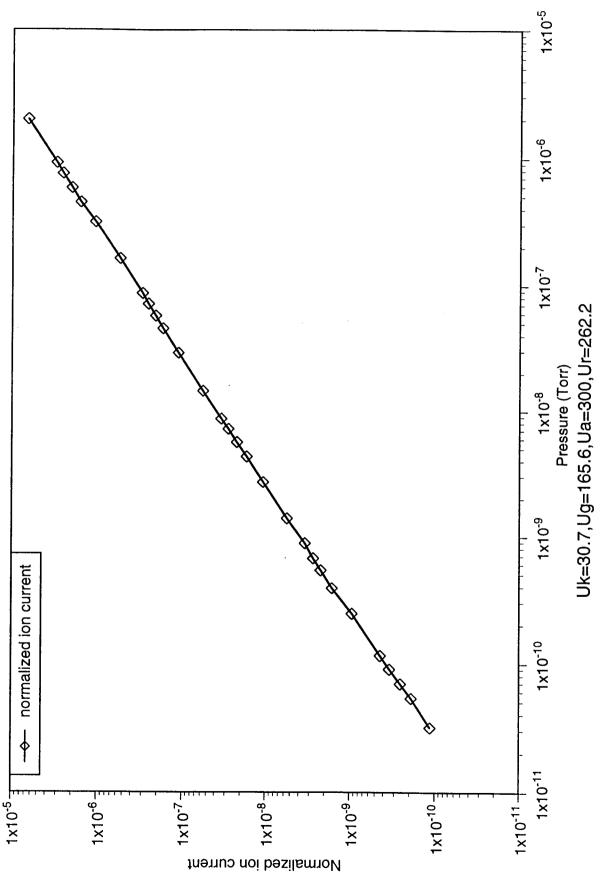
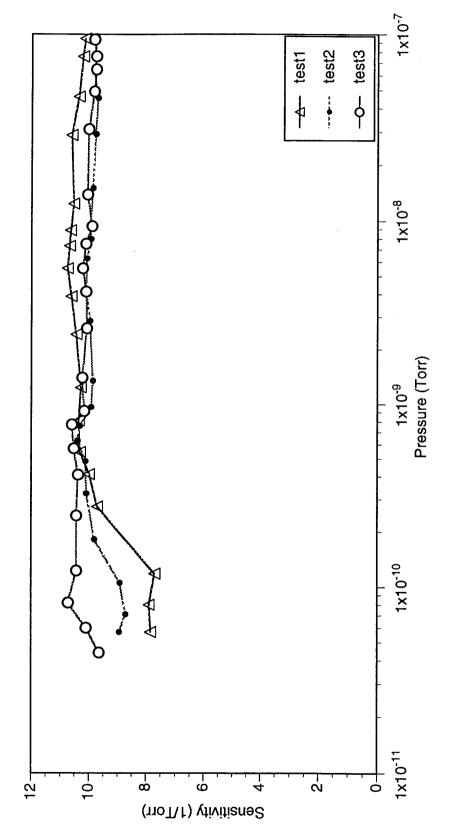


Fig.3 Relationship between pressure and normalized ion current lon currents are normalized to corresponding emission currents

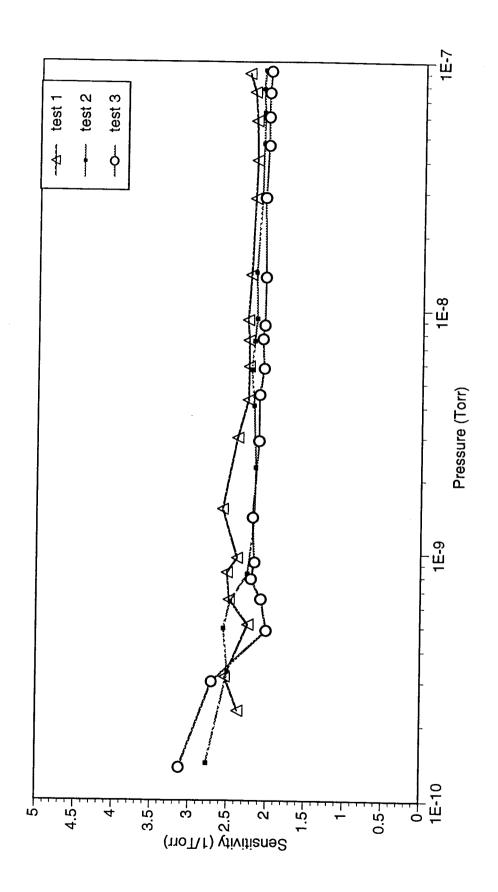
Table 1. Sensitivity comparisons between side FEG and EG

		FEG		EG
Electrode potentials ( Volts )	Ua=220 Uk=98	Ua=250 Uk=120	Ua=500 Uk=305	Ua=220 Uk=100
Nitrogen sensitivity (/Torr)	9	7.4	11.1	8.3 8.3
Helium sensitivity Nitrogen sensitivity	(average of valu	0.157 (average of values from potential 250V to 500V)	)V to 500V)	0.168
Hydrogen sensitivity Nitrogen sensitivity	(average of valu	0.404 (average of values from potential 250V to 500V)	)V to 500V)	0.4



410 /1 e-4 A, 500 / 500 / 300 / 410 /5.5 e-5 A and 500 / 500 / 300 / 405 / 3.3 e-5 A for test1, test2 and test 3 anode voltage / reflector voltage / cathode voltage / gate voltage /emission current = 500 / 500 / 300 /

a. nitrogen



anode voltage / reflector voltage / cathode voltage / gate voltage / emission current = 220 / 220 / 210/ 98 /2.8e-5 A, 220/220 / 215/95 /5.3 e-5 A and 220/220 / 218/93 / 8 e-5 A for test1 test2 and test 3

Fig.4 Sensitivities of the side FEG for nitrogen and hydrogen b. hydrogen

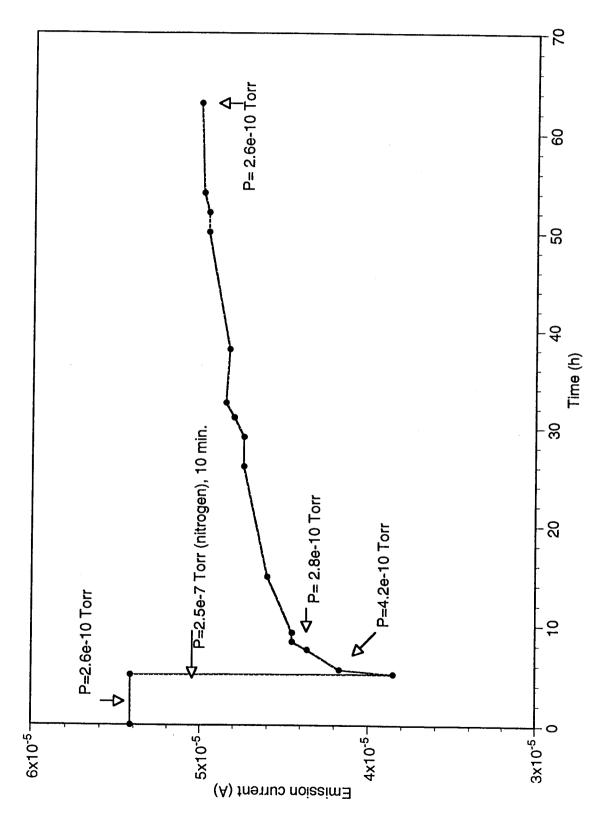
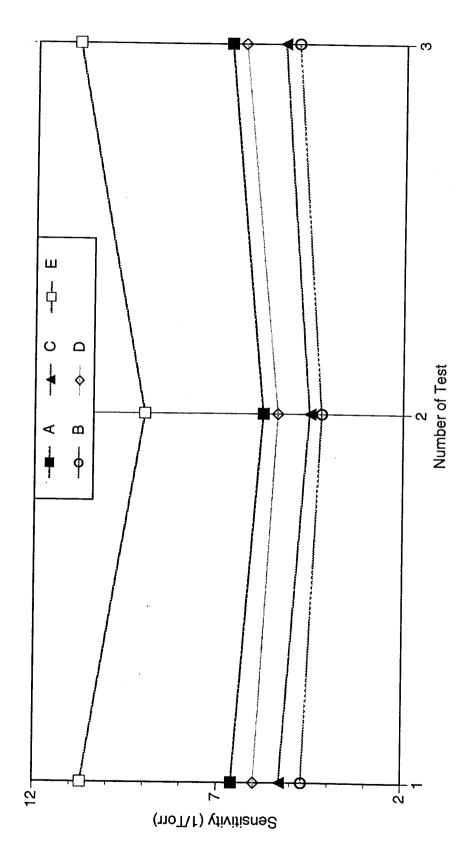
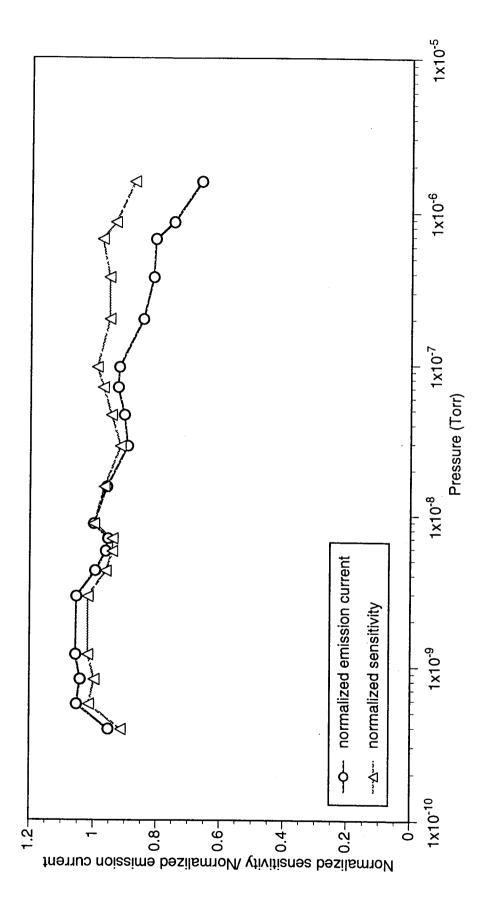


Fig.5 Emission current deterioration after high pressure operation

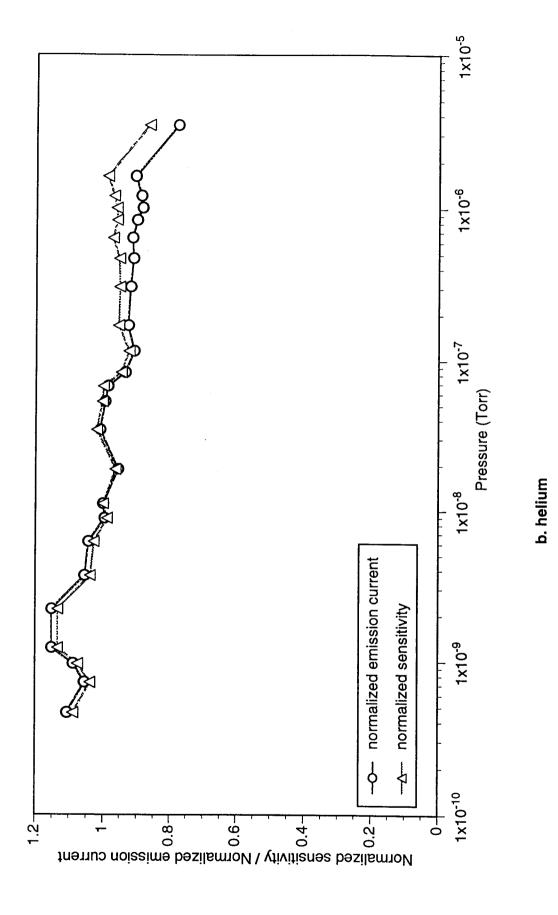


A, B, C, D, and E: Ua/Ur/Uk/Ug/=250/250/115/220, 350/350/225/335, 400/400/258/372, 450/450/289/407, and 500/500/300/410 test 2: after 38 days of tests with nitrogen, helium and hydrogen, 5/22/98 test 3: turning on of the system after 48 hours of inoperation, 6/2/98 test 1: at the begining of using the field emitter, 4/15/98

Fig.6 FEG nitrogen sensitivity changes under different working conditions



a. nitrogen



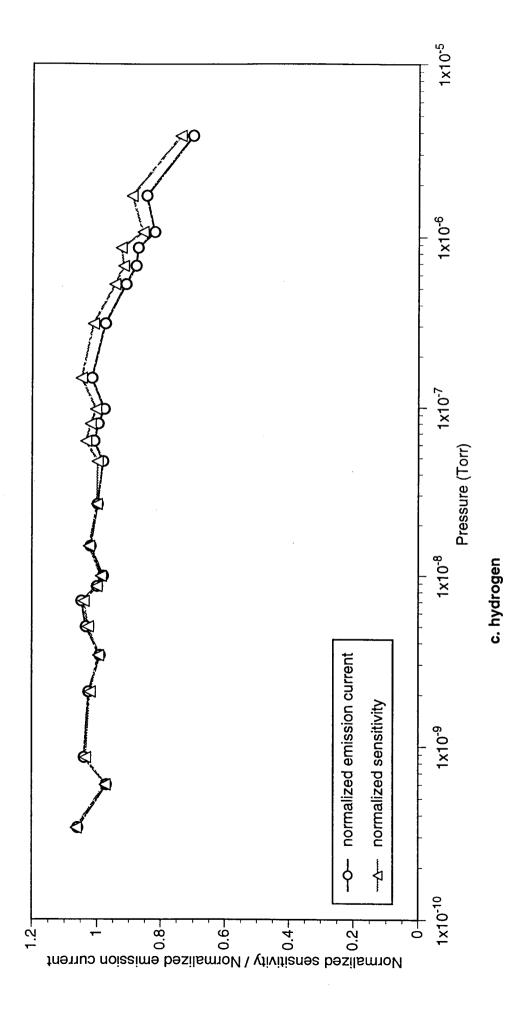


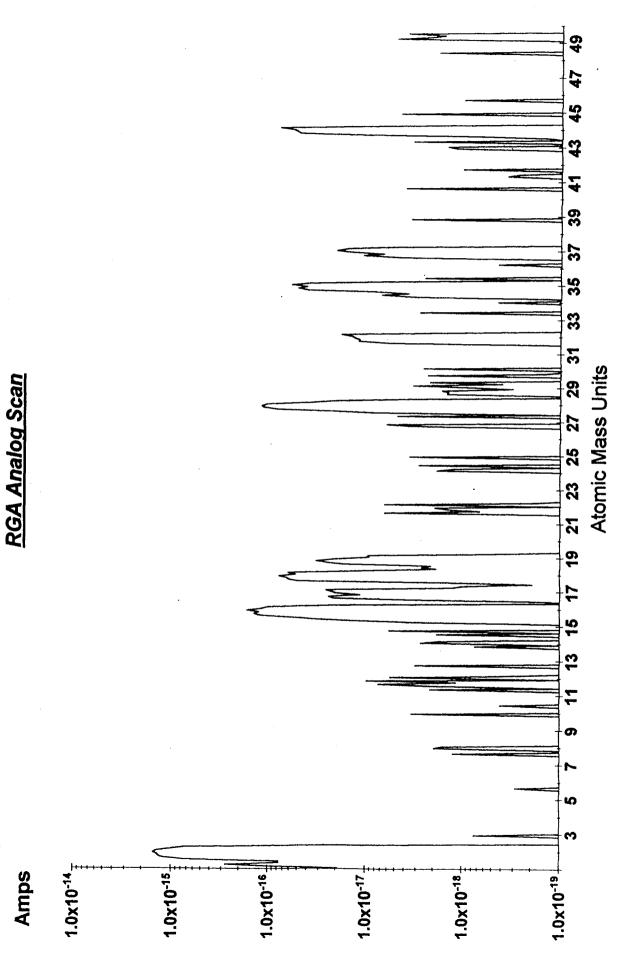
Fig.7 Normalized sensitivities and emission currents for the FERGA

Sensitivities and emission currents are normalized to the values around 1e-8 Torr

FERGA Analog Scan

Amps

a. FERGA



operated under the same pressure and electric parameters. pressure / electron Fig.8 Mass spectra for the FERGA and the hot filament RGA. Both RGAs are current / electron energy = 2.3e-10 Torr / 5.3e-5 A / 80 eV b. the hot filament RGA